

(12) INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(19) World Intellectual Property Organization
International Bureau



(43) International Publication Date
26 April 2001 (26.04.2001)

PCT

(10) International Publication Number
WO 01/28658 A1

(51) International Patent Classification⁷: **B01D 39/20, C04B 35/64**

(74) Agent: VANDERSTERRE, Kees; Corning Incorporated,
SP TI 3 1, Corning, NY 14831 (US).

(21) International Application Number: **PCT/US00/25365**

(81) Designated States (*national*): AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN, CR, CU, CZ, DE, DK, DM, DZ, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, TZ, UA, UG, UZ, VN, YU, ZA, ZW.

(22) International Filing Date:
15 September 2000 (15.09.2000)

English

(25) Filing Language:

English

(84) Designated States (*regional*): European patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE).

(30) Priority Data:
09/418,855 15 October 1999 (15.10.1999) US

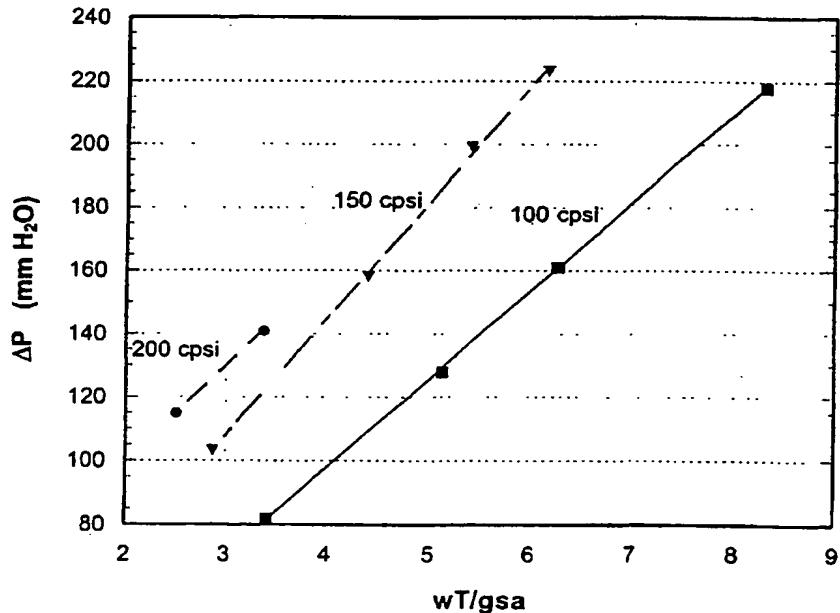
Published:

— With international search report.

(71) Applicant: CORNING INCORPORATED [US/US]; 1
Riverfront Plaza, Corning, NY 14831 (US).

For two-letter codes and other abbreviations, refer to the "Guidance Notes on Codes and Abbreviations" appearing at the beginning of each regular issue of the PCT Gazette.

(54) Title: LOW ASPECT RATIO DIESEL EXHAUST FILTER



WO 01/28658 A1

(57) Abstract: A porous ceramic filter offering both increased thermal mass and controlled exhaust flow performance for the filtration of particulates from diesel exhaust gases comprises a wall-flow honeycomb filter body incorporating a plurality of parallel end-plugged cell channels traversing the body from filter inlet to filter outlet, the wall thickness, cell density, and outer dimensions of the honeycomb being selected to provide a honeycomb bulk density of at least about 0.50 grams/cm³ and an effective honeycomb length-to-diameter aspect ratio not exceeding about 0.9.

-1-

LOW ASPECT RATIO DIESEL EXHAUST FILTER

Background of the Invention

The present invention relates to filters for the removal of particulate material from diesel engine exhaust streams, and more particularly to porous ceramic diesel exhaust filters of improved resistance to melting and thermal shock damage under conditions encountered in diesel exhaust systems.

U.S. 4,420,316 to Frost et al. discusses conventional ceramic wall-flow filter designs suitable for particulate filtration from diesel exhaust gases. As noted in that patent, cordierite ceramic materials of the kinds disclosed in U.S. Pat. Nos. 3,885,977 and 4,001,028 are commonly used for diesel particulate trap filters because they have properties which enable them to withstand the chemical and physical conditions to which they are subjected in the exhaust gas conditions found in diesel engines.

For diesel particulate filtration with these materials, honeycomb structures having cellular densities between about 10 and 300 cells/in² (about 1.5 to 46.5 cells/cm²), more typically between about 100 and 200 cells/in² (about 15.5 to 31 cells/cm²), are considered useful to provide sufficient thin wall surface area in a compact structure. Wall

-2-

thicknesses can vary upwards from the minimum dimension providing structural integrity, about 0.002 in. (about 0.05 mm.), but are generally less than about 0.06 in. (1.5 mm.) to minimize filter volume. A range of between about 5 0.010 and 0.030 inches (about 0.25 and 0.76 mm.) e.g., 0.017 inches, is most often selected for these materials at the preferred cellular densities. Filter aspect ratios (the ratio of filter length to filter diameter) in the range of 0.8-1.2 are usually specified to provide adequate 10 filtration volume in a packaged unit of manageable diameter.

Interconnected open porosity of the thin walls may vary, but is most generally greater than about 25% of thin wall volume and usually greater than about 35% to allow 15 fluid flow through the thin wall longer dimensions.

Diesel filter integrity becomes questionable above about 70% open pore volume; volumes of about 50% are therefore typical. For diesel particulate filtration it is believed that the open porosity may be provided by pores in the 20 channel walls having mean diameters in the range of about 1 to 60 microns, with a preferred range between about 10 and 50 microns.

Filtration efficiencies up to and in excess of 90% of the diesel exhaust particulates (by weight) can be 25 achieved with the described cordierite materials. The filtration of a lesser but still significant portion (i.e. less than 50%) of the particulates may be desirable for other filtering applications including exhaust filtering of smaller diesel engines. Efficiencies, of course, will 30 vary with the range and distribution of the size of the particulates carried within the exhaust stream. Volumetric porosity and mean pore size are typically specified as determined by conventional mercury-intrusion porosimetry.

A significant problem with these conventional filter 35 designs, however, is susceptibility to damage during the

-3-

required filter regeneration cycling. In conventional diesel exhaust filtration systems, exhaust gas flow rate and thus engine operating efficiency are affected by the increasing pressure drop across the filter caused by the
5 accumulation on the filter of collected carbonaceous exhaust particulates (soot). Eventually, this pressure drop becomes unacceptable and regeneration of the filter becomes necessary. In conventional systems, the regeneration process involves heating the filter to
10 initiate combustion of the soot, a process that is highly exothermic and, if uncontrolled, produces temperature spikes that can thermally shock and crack, or even melt, the filter structure.

This problem has been recognized and several
15 approaches to address it have been proposed. One such approach involves the careful selection of filter design parameters, as reported by H. Mizuno, J. Kitagawa, and T. Hijikata in "Effect Of Cell Structure On Regeneration Failure Of Ceramic Honeycomb Diesel Particulate Filter",
20 SAE Paper No. 870010 (1987). Within a filter cell density range of about 100-200 cells/in², Mizuno et al. found that increasing filter bulk density through increases in filter wall thickness and cell density effectively increased filter heat capacity, thereby moderating the peak filter
25 temperatures encountered during filter regeneration. However, the thicker-walled filters exhibited higher pressure drops than standard filters, and in addition appeared to reach higher exhaust back-pressure levels in shorter times and at lower soot loadings than filters with
30 thinner walls. These disadvantages suggested that an optimal combination of soot capacity and regeneration performance resided in a relatively narrow range of filter geometry characterized by filter wall thicknesses in the 0.012-0.017 inch range and cell densities in the 100-200
35 cells/in² range.

-4-

Notwithstanding these findings, cordierite diesel exhaust filters produced in accordance with these guidelines, including standard commercial filters with cell densities of 100 cells/in and wall thicknesses of 5 0.017 inches, continue to experience thermal failures with unacceptable frequency in use and under test conditions approximating those found in diesel exhaust systems. Typical failures include filter cracking resulting from thermal stress and shock, as well as occasional melting 10 resulting when large soot deposits undergo rapid and uncontrolled combustion.

Summary of the Invention

The present invention provides porous ceramic particulate exhaust filters offering improved 15 configurations that are significantly more resistant to thermal cracking and melting damage under typical diesel exhaust conditions than current filter designs. At the same time, the filters of the invention retain favorable 20 initial exhaust gas back-pressures and promise resistance to soot-induced back-pressure buildup at least equivalent or superior to the best ceramic diesel particulate filter designs currently available.

In particular, the invention provides a ceramic 25 filter for trapping and combusting diesel exhaust particulates which includes an end-plugged porous ceramic honeycomb structure with both increased thermal inertia and reasonable pressure drop in a diesel exhaust system. The increased thermal inertia or mass required in the 30 filter is obtained by increasing the bulk density of the honeycomb structure to a value of at least 0.50 grams/cm³. At the same time, initial filter pressure drop and the rate of back-pressure increase during use are regulated by reducing the aspect ratio, but not the filtration surface 35 area, of the filter. These changes in filter design

-5-

provide filters with significantly increased thermal inertia that still retain particulate-free or "clean filter" exhaust gas pressure drops of 160 cm (water column) or less at conventional diesel exhaust gas flow rates.

5 Filter performance characteristics such as described can readily be secured in porous ceramic wall flow filter bodies of conventional end-plugged honeycomb structure and porosity, whether formed of cordierite or of other
10 similarly porous but thermally durable ceramic materials, provided that the geometry of the filter bodies is maintained within specified ranges. Specifically, the filter bodies of the invention will be characterized by a honeycomb channel wall thickness of at least 0.016 inches
15 and channel or cell densities of at least 150 cells/in², but with effective filter length-to-diameter aspect ratios not exceeding about 0.9, more typically about 0.2-0.85.

For purposes of the present description filter bulk density is simply the quotient of filter weight divided by
20 the overall volume of the filter (i.e., the volume determined from the exterior dimensions of the filter). The filter aspect ratio is the ratio of the length of the filter along the filter axis parallel to the direction of the end-plugged channels to the effective diameter of the
25 filter. The effective diameter of a filter of non-circular cross-section is taken to be the diameter calculated from the area of the filter cross-section perpendicular to the channel axis if converted to circular shape. The filter of course need not be circular in
30 cross-section but may be of circular, oval, square, rectangular or any other cross-sectional shape that may be dictated by the particular diesel exhaust system design selected for use.

-6-

Brief Description of the Drawings

The invention may be further understood by reference to the drawings, wherein:

Fig. 1 presents exhaust flow back-pressure data for a series of porous ceramic filters of constant porosity but differing cell density and cell wall thickness;

Fig. 2 plots filter back-pressure versus filter aspect ratio for a series of porous ceramic filters of otherwise identical geometry, and

Fig. 3 compares the relative rates of filter back-pressure increase due to particulate accumulation for a series of porous ceramic filters of identical volume and cell design but differing aspect ratio.

15 Detailed Description

Porous ceramic diesel filters of the type herein described are referred to as "wall flow" filters since the flow paths resulting from alternate channel plugging require the fluid being treated to flow through the porous ceramic cell walls prior to exiting the filter. As the art is well aware, the primary cause of pressure drop for a wall flow gas filter arises from the flow resistance associated with the passage of the gas through the wall. This resistance is affected by the porosity and thickness of the wall and, for the filter as a whole, the total or aggregate wall surface area available for filtration. If wall porosity is fixed, then wall flow resistance is found to be directly proportional to the wall thickness and inversely proportional to the aggregate wall area, referred to as the filter geometric surface area (GSA).

Fig. 1 of the drawing presents representative back-pressure data for several conventional porous cordierite diesel filters of constant porosity but differing cell density and cell wall thickness, taken from the

- 7 -

literature. In that graph, filter back-pressure ΔP , shown on the vertical graph axis, is plotted against filter wall thickness and geometric surface area (GSA) on the horizontal axis. More specifically, the horizontal scale indicates the ratio of filter wall thickness to filter geometric surface area (wT/gsa) for each filter design tested. Three different cell densities are plotted, but all filters are of the same overall dimensions.

The pressure testing was conducted at a constant gas flow rate of about $1.3\text{m}^3/\text{minute}$ (gas volumes as measured at 1 atmosphere and 20°C .). The observed differences in flow resistance are attributable principally to the differences in wall thickness and geometric surface area. As expected, the data reflect a linear dependence of filter back-pressure on the ratio of filter wall thickness to filter geometric surface area for these filters.

The data in Fig. 1 do not reflect clearly the effects of laminar flow rate and gas expansion/contraction on filter back-pressure. That is, the pressure drop across a honeycomb filter substrate also depends on the resistance to laminar flow of gas down the cell channels and, as a second order effect, the extent of gas contraction and expansion occurring as the gas traverses the cellular structure.

One manifestation of this effect is illustrated in Fig. 2 of the drawing, which is a plot of filter back-pressure (Pressure Drop in cm of H_2O) versus filter length (in inches) for a series of filters of identical wall thickness, cell density and volume, but varying aspect ratio. Although the geometric surface areas of these filters are essentially equivalent, such that wall flow resistance is nearly invariant, significant variations in back-pressure are observed. These are considered to arise from fluid flow and gas compression effects.

- 8 -

Filters provided in accordance with the present invention take advantage of these hydraulic effects to provide filters with higher bulk density and therefore higher thermal inertia than previously considered practical. These designs are not only more resistant to undue heating during the regeneration cycle than the commercial designs of the prior art, but also offer favorable initial pressure drop and resistance to plugging equivalent to or better than those of conventional filters.

This combination of properties is secured through the adoption of specific low-aspect-ratio filter designs that more effectively control hydraulic back-pressure effects encountered in actual use. Particularly preferred filter geometries offering useful regeneration heat moderation and low pressure drop are those with filter bulk densities in the range of 0.60-0.85, cell densities in the range of about 200-300 cells/in², wall thicknesses in the range of about 0.016-0.030 inches, and aspect ratios within the range of about 0.2-0.85. Within this region of honeycomb design, clean filter pressure drops well below 160cm (water column) at exhaust gas flow rates of 1.3 m³/min. or lower can readily be achieved.

A further and previously unreported advantage of these low aspect ratio filter designs appears to be a reduction in the frequency of filter regeneration that is required in use. This advantage results from the fact that the increases in filter back-pressure arising from the accumulation of particulate material on the walls of the filter inlet channels occur more slowly in low-aspect-ratio filters than in filters of conventional design. Thus the intervals between required filter regeneration cycles can apparently be considerably extended through the adoption of a low-aspect-ratio design.

- 9 -

For new filters, or during an initial period of filter use interval immediately following a complete filter regeneration, pressure drop across the filter increases approximately linearly over time with the steady accumulation of new particulate material on the inlet channel walls of the filter. However, the rate of increase during this initial use interval, and over much of the ensuing normal filter use interval, is found to be highly dependent on the aspect ratio of the filter, other geometric variables such as wall thickness, cell density, and filter volume being constant.

Fig. 3 of the drawing illustrates this effect in a graph showing the relative rate of back-pressure increase per unit of time as a function of filter aspect ratio for four different filter shapes. The back-pressure increase rate (ΔP Increase per hour), indicated on a relative scale on the vertical axis, is plotted against the filter aspect ratio factor $1/D^2$ (inverse Diameter squared) on the horizontal axis of the graph. The filters tested were all of the same cell density, wall thickness and volume. The strong dependence of filter back-pressure build-up rate on the filter aspect ratio is shown by the approximately linear dependence of that rate on the inverse square of the filter diameter.

A representative example of a filter design provided in accordance with the invention, and the performance advantages resulting therefrom, may be better understood from the following illustrative example, which is not intended to be limiting.

Example

A cylindrical cordierite ceramic honeycomb of 5.66 inch diameter and 4.7 inch length, having about 200 cells per square inch of honeycomb frontal surface area

-10-

traversing its length and with a 0.020 inch wall thickness, is provided by first extruding a plasticized mixture of clay, talc, alumina and graphite through a honeycomb extrusion die to form a green honeycomb. The 5 green honeycomb is then dried and fired to produce a fired honeycomb having a thermal expansion coefficient of about $5 \times 10^{-7}/^{\circ}\text{C}$. and interconnecting wall porosity of about 50% by volume. The average pore size in the channel walls is about 13 μm .

10 Half of the channels of the fired honeycomb are plugged at their inlet ends with a ceramic sealing compound in a checkerboard plugging pattern, and the remaining channels are similarly plugged at their outlet ends. The plugged honeycomb is thereafter fired again to 15 cure the ceramic seals and permanently plug the channels to form a wall-flow filter body. The result is a plugged, fired honeycomb wall filter with a length-to-diameter aspect ratio of about 0.83 and a open frontal area providing a filter bulk density of 0.62 g/cm³ of filter 20 volume.

The mounting of a fired honeycomb filter produced as described involves positioning the filter snugly within a cylindrical steel filter enclosure with a refractory resilient mat disposed between the filter sidewall and the 25 wall of the enclosure. The ends of the enclosure may then be provided with inlet and outlet cones for channeling exhaust gas into and through the alternately plugged channels and porous walls of the filter.

To preserve the reduced aspect ratio of this filter 30 and insure that the entire volume and wall structure thereof remains available for exhaust filtering, the honeycomb mounting in this particular case will be carried out in such a way that the front and rear faces of the honeycomb remain entirely unobstructed. Alternatively,

-11-

the filter could be extruded and fired at slightly larger cylinder diameter, e.g., a fired outer diameter of about 6.4 inches, and then conventional edge gasketing and retention sleeves used to fix the honeycomb in the
5 enclosure without concern for the resulting drop filter volume and wall surface area.

Assuming a test exhaust gas flow rate of $1.3\text{m}^3/\text{minute}$ (Standard gas temperature and pressure), extrapolation from "clean filter" pressure drop data reported in the
10 literature for filters of the same cell density (but with higher aspect ratios and lower wall thicknesses and bulk densities), indicate an initial pressure drop of about 13 cm water column for this filter at the same exhaust flow rate. This initial pressure drop is comparable to prior
15 art filters of higher aspect ratio, but the bulk density of the present filter is significantly higher than the prior art filters, indicating that the resistance of the filter to temperature spiking during the regeneration cycle will be significantly improved.

Another significant performance advantage of this filter design is expected to reside in its reduced rate of pressure drop increase as soot is collected by the filter in actual use. As indicated in Fig. 3 of the drawing, the
20 rate of pressure drop increase with soot accumulation in these filters is expected to depend strongly on the aspect ratio of the filter, with low aspect filters offering a significant performance advantage over higher aspect filters of the same volume, cell density and wall thickness. Based on the magnitude of this effect as
25 reflected in the data reported in Fig. 3, the rate of pressure drop increase with soot accumulation for the 0.83 aspect ratio filter of this example will be approximately 25% less than the rate of increase for a 1.2 aspect ratio part of the same volume, cell density and wall thickness.
30 And, hydraulic principles indicate that the filter of the
35

-12-

example will have a slightly greater soot holding capacity before regeneration is required than the higher aspect filters of the prior art.

Additional examples of diesel particulate filters covering a range of aspect ratios, wall thicknesses and bulk densities are reported in Table 1 below. Several Standard filter designs taken from the literature, along with representative examples of Low aspect ratio, High bulk density filter designs provided in accordance with the invention, are included. Recorded for each of the filters reported in the Table are the size, aspect ratio, wall thickness and bulk density of the filters, along with estimated initial pressure drop and back-pressure increase data where available.

-13-

Table I - Filter Designs

	Standard Filters				High-Density, Low Aspect Ratio Filters				
	A	B	C	D	E	F	G	H	I
Diameter (inches)	5	5	5	5	5	5.7	6	6.5	7
Length (inches)	6	6	6	6	6	4.6	4.2	3.6	3.1
Wall Thickness (inches)	0.017 4.32	0.012 3.05	0.015 3.81	0.017 4.32	0.022 5.59	0.020 5.68	0.025 6.35	0.030 7.62	0.020 5.08
Cell Density (cells/in ²)	100 15.5	200 31	200 31	150 15.5	150 15.5	200 31	250 31	200 31	300 46.5
Aspect Ratio	1.2	1.2	1.2	1.2	1.2	0.82	0.72	0.54	0.43
Filter Volume (inches ³)	118	118	118	118	118	118	118	118	118
Filter G.S.A. (inches ²)	3909	5531	5248	4567	4214	4786	4508	3847	5345
Initial Pressure Drop (cm H ₂ O)	128	115	141	158	223	131	142	160	110
Open Frontal Area	69%	69%	62%	63%	53%	51%	37%	33%	43%
Filter Bulk Density	0.39	0.39	0.48	0.47	0.59	0.61	0.80	0.84	0.72
Projected Plugging rate (cm H ₂ O/hr.)	140	70	100	130	200	105	100	95	80

-14-

As is evident from a study of the data in Table 1,
the low aspect ratio filters of the invention offer
significantly increased thermal mass (bulk density) when
compared with the density levels employed in the prior
5 art. Yet the filter back-pressures and pressure drop
increase rates expected with these filters are at least
equivalent to and typically significantly better than
those measured for prior art filters. Thus substantial
reductions in the incidence of regeneration damage, and
10 useful improvements in soot capacity and soot plugging
rates, are anticipated.

-15-

I claim:

1. A ceramic filter for trapping and combusting diesel exhaust particulates comprising an end-plugged porous ceramic honeycomb structure having:

5 a honeycomb bulk density of at least about 0.50 grams/cm³; and

an effective length-to-diameter aspect ratio not exceeding about 0.9.

10

2. A ceramic filter in accordance with claim 1 which has a particulate-free exhaust gas pressure drop not exceeding about 160 cm H₂O at an exhaust gas flow rate of 1.3 m³/min.

15

3. A ceramic filter in accordance with claim 2 which is composed substantially of cordierite.

20

4. A diesel exhaust particulate filter comprising a plugged, wall-flow honeycomb filter body composed of porous ceramic material and comprising a plurality of parallel end-plugged cell channels traversing the body from a frontal inlet end to an outlet end thereof, wherein:

25

the honeycomb body has a honeycomb bulk density of at least about 0.60 grams/cm³, a cell density of at least 150 cells per square inch of frontal inlet area, a cell wall thickness of at least about 0.016 inches, and an effective length-to-diameter aspect ratio not exceeding about 0.9.

30

5. A diesel exhaust particulate filter in accordance with claim 4 which is composed substantially of cordierite.

-16-

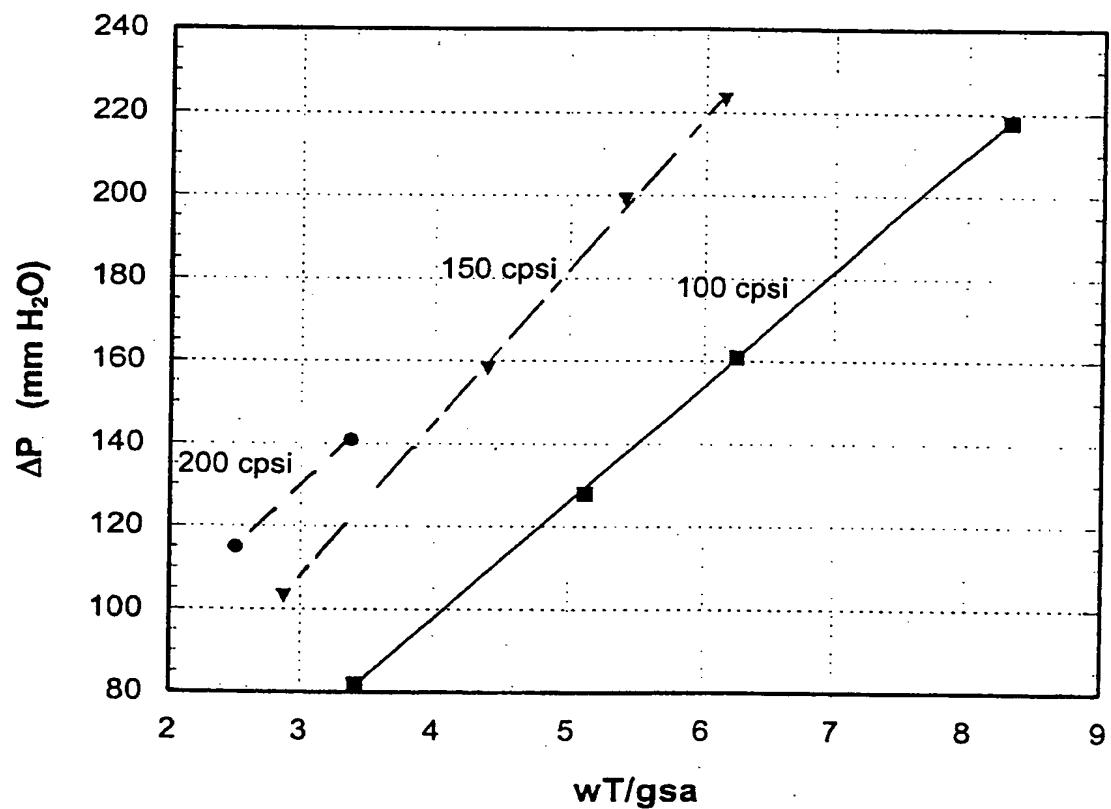
6. A diesel exhaust particulate filter in accordance with claim 5 which has an effective length-to-diameter aspect ratio in the range of about 0.2-0.85.

5 7. A diesel exhaust particulate filter in accordance with claim 6 which has a filter bulk density in the range of 0.60-0.85, a cell density in the range of about 200-300 cells/in², and a cell wall thickness in the range of about 0.016-0.030 inches.

10

8. A diesel exhaust particulate filter in accordance with claim 7 having a clean filter pressure drop not exceeding 160cm (water column) at an exhaust gas flow rate not exceeding 1.3 m³/min.

1/3

**Fig. 1**

2 / 3

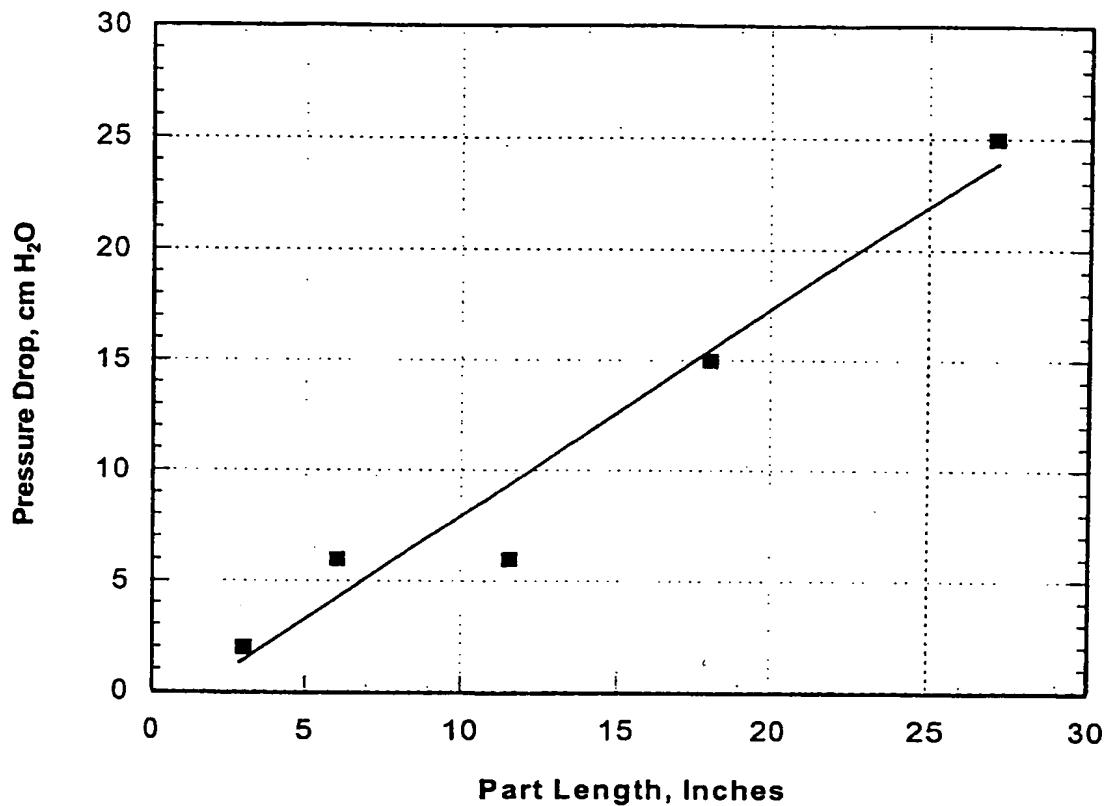


Fig. 2

3/3

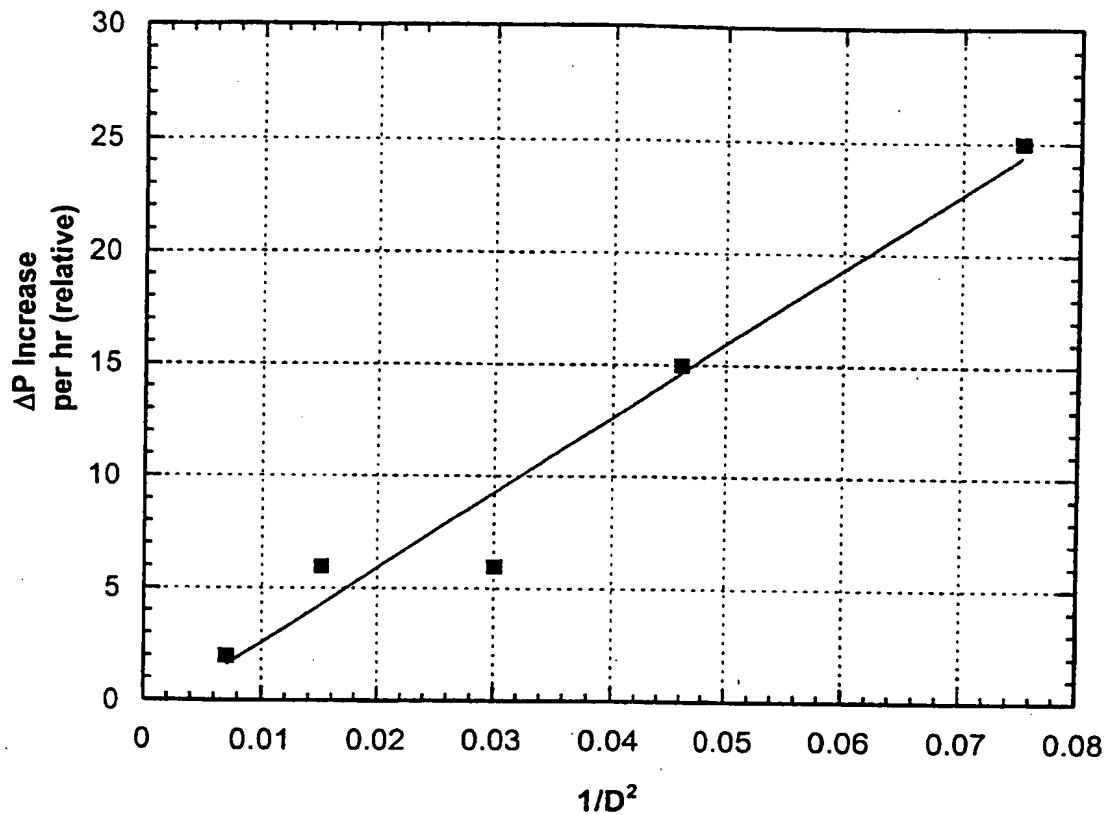


Fig. 3

INTERNATIONAL SEARCH REPORT

International application No. PCT/US00/25365

A. CLASSIFICATION OF SUBJECT MATTER

IPC(7) :B01D 39/20; C04B 35/64
US CL :55/523, DIG. 30

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

U.S. : 55/523, DIG. 30; 60/311; 210/510.1

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

NONE

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

NONE

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	US 6,101,793 (NAGAI et al.) 15 August 2000.	1-8
A	US 5,549,725 A (KASAI et al.) 27 August 1996.	1-8
A	US 5,069,697 A (HAMAGUCHI et al.) 03 December 1991.	1-8
A	US 4,869,944 A (HARADA et al.) 26 September 1989.	1-8
A	US 4,857,089 A (KITAGAWA et al.) 15 August 1989.	1-8

Further documents are listed in the continuation of Box C. See patent family annex.

* Special categories of cited documents:	"T"	later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention
A document defining the general state of the art which is not considered to be of particular relevance	"X"	document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone
E earlier document published on or after the international filing date	"Y"	document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art
L document which may throw doubt on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)	"&"	document member of the same patent family
O document referring to an oral disclosure, use, exhibition or other means		
P document published prior to the international filing date but later than the priority date claimed		

Date of the actual completion of the international search
28 NOVEMBER 2000

Date of mailing of the international search report

04 NOV 2001

Name and mailing address of the ISA/US
Commissioner of Patents and Trademarks
Box PCT
Washington, D.C. 20231
Facsimile No. (703) 305-3230

Authorized officer
DUANE S. SMITH
Telephone No. (703) 308-0651

Jean Proctor
Paralegal Specialist